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Measurements on the contribution of thermal lattice defects to structural energy and expansion in pure metals

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With 10 figures in the text

1. Introduction

Recent measurements on very pure, coarse-grained silver [1] have shown that the development of certain thermal defects is slow enough to be studied by means of isothermal calorimetry. When the specimens were heated from room temperature to various temperatures in the range 160° to 380°C, it was found that the curve of energy absorption as a function of time during the approach to equilibrium at the new temperature had an exponential tail with a time constant of the order of a few hours. The energy of the formed defects calculated from these curves increased with temperature as

$$Q = Q_0 \cdot \exp\left(-\frac{H}{RT}\right) \tag{1}$$

where H is a heat of formation, Q_0 is a constant, which was found to increase with increasing purity and grain size of the specimens, and R is the gas constant. Experimentally obtained values for 99.999 % silver were $H = 4700 \pm 700$ cal/mole or 0.20 ± 0.03 eV and $Q_0 = 80$ to 650 cal/mole, the highest value being obtained for the most coarse-grained specimen. Another characteristic feature for the process was the observation that the time constant for the exponential heat-absorption curves decreased very little with increasing temperature. In fact the energy of activation derived from the temperature dependence of the time constants was only about 1000 cal/mole corresponding to 0.04 eV. The discussion of different possibilities indicated that the most probable interpretation of those results was that they were due partly to the unidentified defects causing the increase of the structural energy discussed by Borelius [2, 3]. The temperature dependence of the structural energy revealed the existence of two different kinds of lattice disturbances. One of these, representing the greater part of the structural energy, had an energy of formation of about 0.2 eV for the noble metals. The nature of the corresponding thermal defects could not be established. The other part of the structural energy, which appeared with an energy of formation of about 1 eV, was identified with lattice vacancies. Borelius has also tried to divide the thermal expansion into a vibrational and a structural part [4]. The analysis showed that the structural part could be separated in two thermally activated effects, similar to what was obtained in [2] and [3].

The present paper will describe the results of further measurements of the structural energy for gold, aluminium and molybdenum. These measurements confirm the earlier results for silver. For gold, in addition, the corresponding changes in the thermal expansion have been studied, using an isothermal technique. When heating from room temperature to a higher temperature the thermal equilibrium value of the dilatation was approached exponentially with time in a manner similar to the calorimetric case. The temperature dependence of the effects was also consistent in both cases.

2. Calorimetric measurements

Method and results

For the calorimetric measurements the same isothermal method was used as described in [1]. The rate of change of energy was studied as a function of time. Temperature differences between the specimen and the thermostat could be followed down to some tenthousandths of a degree. The range of measurements was from 160° to 480°C.

Three metals of high purity were used, namely aluminium of 99.997 % purity, gold 99.998 % and 99.98 % (denoted I and II, respectively), and molybdenum 99.992 %. All metals except gold II were spectrographically standardised substances from Johnson, Matthey & Co. The analyses do not, however, include a solubility of gases such as hydrogen, nitrogen, and oxygen. The following solubilities of hydrogen can be expected: 4×10^{-7} wt % at 400°C for Al [5] and 0.9×10^{-5} wt % at 500°C for Mo [6]. Nitrogen and oxygen seem to be practically insoluble in these metals. For gold no solubilities of these gases have been reported. The metals were annealed *in vacuo* immediately below the melting point and then slowly cooled to room temperature. Aluminium and gold had a grain-size of about 1 mm, while for molybdenum the grain-size was about 0.1 mm. The mass of the specimens must necessarily be relatively large (half a mole) as the heat effects to be studied were small. In order to avoid an eventual deterioration of the samples, they were enclosed in thin-walled, evacuated Pyrex containers during the pre-annealings and the measurements.

The experimental procedure was in principle the same as described in [1]. Thus the specimens were heated from a lower temperature to the thermostat temperature, or they were quenched from a higher temperature directly to this temperature. In the first case the metal was pre-annealed for some days at the thermostat temperature and was then cooled in air to room temperature and aged there for some days. The specimen was then rapidly heated back to the thermostat temperature in an external oven and inserted in the thermostat for observation of the energy changes. The rate of change of energy versus time at different temperatures is shown in Figs. 1–3. Zero on the time scale is the moment when the specimen has just reached the measuring temperature. Due to a slight disturbance of the thermal equilibrium of the termostat when the specimens were inserted into it, the first hour of the run escapes accurate measurements. From Figs. 1–3 it is evident that the heat-absorption in the specimens increases with temperature. As in the earlier measurements [1] the rate of energy absorption during the approach to equilibrium at the measuring temperature showed an exponential tail of the form $dQ/dt = (dQ/dt)_0 \exp(-t/\tau_{\mathrm{cal}})$. The time constant τ_{cal} from the calorimetric measurements was of the order of some hours. $(dQ/dt)_0$ represents the initial value of the rate of change of energy as obtained by exponential extrapolating of the experimental curves. The energy Q is obtained by

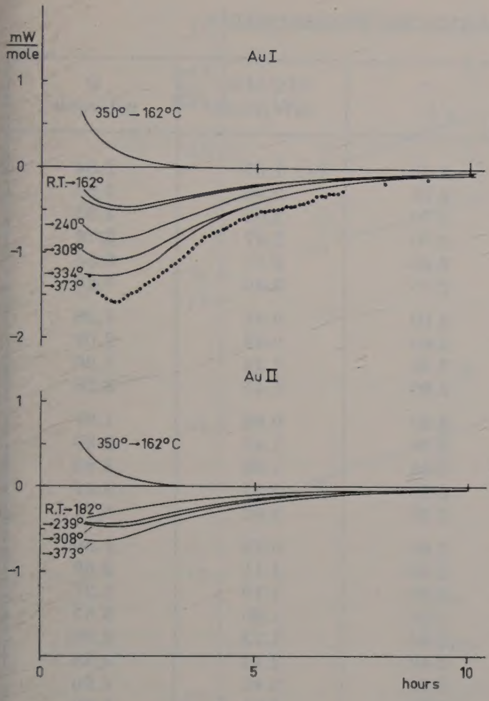

Fig. 1.

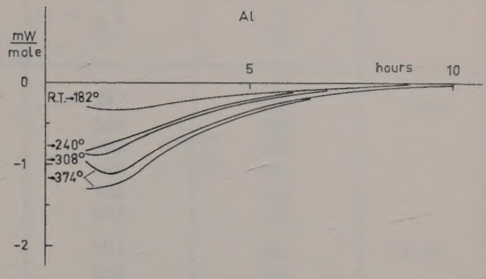

Fig. 2.

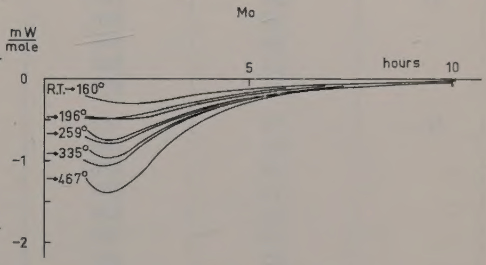

Fig. 3.

Figs. 1–3. Rate of energy absorption and release versus time for measurements on Au, Al and Mo after different pre-annealings. The scattering of the observed values is shown for one measurement.

integration of these curves from $t = 0$. By the exponential extrapolation we do not account for the initial decrease of the rate of change of energy which in most cases in Figs. 1–3 gives rise to a minimum in the curves. It seems, however, probable, especially from the measurements in [1], that this part of the curves is due to disturbances owing to the experimental technique used. The values of τ_{cal}, $(dQ/dt)_0$ and Q obtained from Figs. 1–3 are given in Table 1. T_a represents the ageing temperature and T_m the measuring temperature.

In the measurements in Figs. 1–3 the specimens were cooled in air to T_a, which took about 10 minutes. The ageing time was then two days. Variation of the cooling time in the range 1 minute to 8 hours, however, had no great effect on the amount of energy taken up. Although the cooling can be done more quickly it is questionable whether it is possible to quench in the effect on account of the relatively large mass of the calorimetric specimens. The ageing time seems not to be very critical either, as it was found that almost the same amount of energy was absorbed when the specimens were measured after they had just reached room temperature as in the case when they were aged for some days at T_a before being measured.

The energy which the specimens take up to reach equilibrium increases with the difference between measuring and ageing temperature. For Ag the energy was found to increase exponentially with temperature according to eq. (1). In Fig. 4 the

Table 1. Results of the calorimetric measurements.

	T_a °C	T_m °C	τ h	$(dQ/dt)_0$ mW/mole	Q cal/mole
Au I	26	182	3.09	1.07	2.84
	26	182	3.17	0.96	2.62
	26	240	2.70	2.02	4.52
	26	308	2.70	2.47	5.75
	25	334	2.40	3.17	6.55
	25	373	2.75	3.46	8.20
Au II	26	182	3.10	0.51	1.36
	26	239	2.60	0.93	2.09
	26	308	2.50	1.24	2.66
	26	373	2.60	1.45	3.26
Al	26	182	3.30	0.66	1.87
	26	240	2.70	1.42	3.30
	26	308	2.35	1.89	3.83
	25	374	2.20	2.77	5.27
	26	374	2.35	3.02	6.11
Mo	24	160	2.65	0.79	1.80
	24	196	2.80	1.11	2.69
	24	196	2.50	1.10	2.37
	25	259	2.50	1.66	3.57
	25	259	2.40	1.73	3.59
	25	335	2.40	2.12	4.38
	25	335	2.20	2.42	4.60
	27	467	1.95	3.74	6.30

logarithm of the Q-values from Table 1 have been plotted versus the reciprocal of the absolute temperature. Obviously the same exponential relation is valid also for the present measurements. For the following discussion we prefer, however, to describe the experimental results by the equation

$$Q = Q_c \cdot \exp\left[-\frac{H_{\mathrm{cal}}}{R}\left(\frac{1}{T_m} - \frac{1}{650}\right)\right] \tag{2}$$

which is approximately valid in the range of measurements. Q_c is the structural energy absorbed when heating from room temperature to 650°K. Q_c and the energy of formation H_{cal}, obtained from the slope of the lines in Fig. 4, have been collected in Tables 3 and 4 for the different metals. The maximal error of the values is about 20 %. In Tables 3 and 4 the results from earlier measurements on 99.999 % Ag [1] have also been included. Three specimens of different grain size were used in that case: A, 0.3 mm; B, 1 mm; and C about 1 cm. The results in Tables 3 and 4 will be discussed in section 4.

The influence of temperature on the time constants of the exponential heat-absorption curves is very small. In fact the energies of activation as derived from the log $\tau - 1/T$ diagram in Fig. 5 are only about 1000 cal/mole (0.04 eV) to within about 30 %.

The effect of impurities on the absorbed energy is consistent with the results in [1]. It is thus obvious from Fig. 4 that the purer gold specimen (I) takes up more energy than the less pure (II), the ratio being about 2:1.

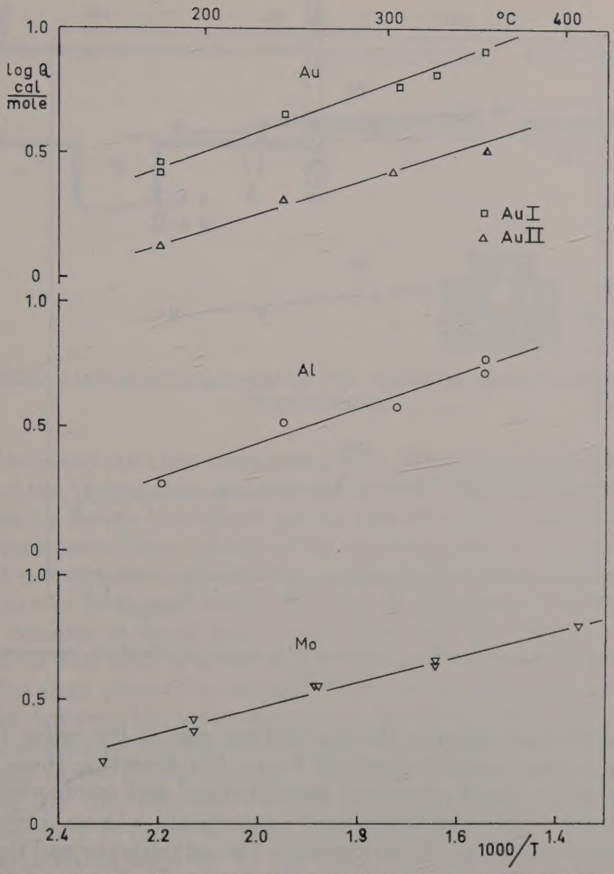

Fig. 4. Logarithms of absorbed energies versus reciprocals of absolute temperatures for Au, Al and Mo.

In the case of gold some experiments were also made in which T_a was higher than T_m. When the cooling of the specimens was interrupted, a small isothermal heat evolution was observed as shown in the upper curves of I and II in Fig. 1, where the specimens have been taken from 350° to 162°C. At higher measuring temperatures we have not obtained any reliable results for a heat treatment of this kind. Probably the process is too rapid here to be measured with the present method. The observed energy release is about 20 % of the quantity $(Q_{350^\circ} - Q_{162^\circ})$ for Au. The difference in energy is probably lost during the cooling. The heat release does not follow simple first order kinetics. The dependence of this effect on temperature has not been investigated.

3. Expansion measurements

Method

From a comparison of the results of the calorimetric measurements and the computations of Borelius it could be expected that the structural part of the linear

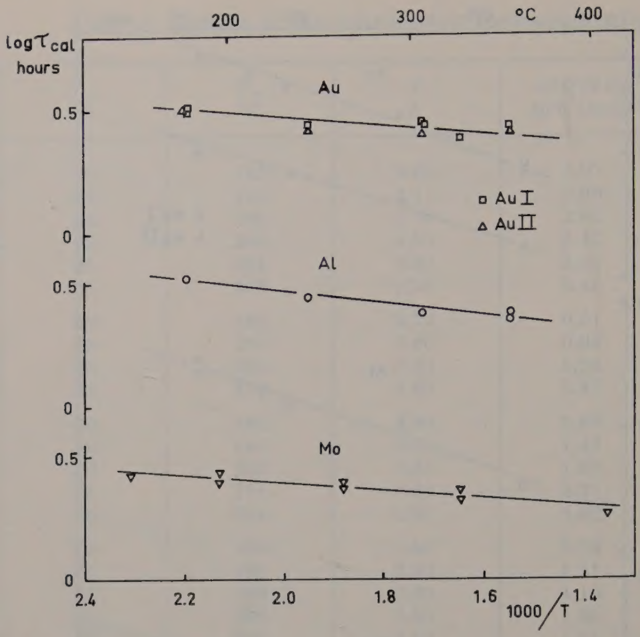

Fig. 5. Logarithms of time constants τ_{cal} versus reciprocals of absolute temperatures for Au, Al and Mo.

expansion due to the formation of thermal defects was of the order 10^{-5}, the whole effect developing in the course of about 20 hours. For detecting these small effects a sensitive dilatometric method possessing good thermal and mechanical stability was needed. The method which was found most appropriate was an interferometric one which in principle is the usual Fizeau method. The advantages and the limitations of this method have been discussed in a paper by Austin [7]. For our purpose the small dimensions and the simple construction of the apparatus were of importance, as it was to be enclosed in the relatively small core of a good thermostat. A restriction in the use of the method is the necessity of correcting the refractive index of air for variations in pressure and temperature. The influence of pressure was eliminated by making the measurements in vacuum, whereby the error due to an eventual oxidation of the specimens was also avoided.

The apparatus used consists in principle of an interferometer inserted in a thermostat, which can be evacuated. A schematic view of the arrangement is shown in Fig. 6*a*.

The interferometer (1) is mounted on three horizontal bars of stainless steel (2) of a length of about half a meter. The bars are attached to a brass tube containing a window (3), a Pt-PtRh thermocouple (4) by which the temperature of the interferometer can be determined, connections for the vacuum pumps (5), and arrangements for measuring the pressure. The brass tube is firmly mounted on a vibrationless support. The thermostat is of a type described by Borelius *et al.* [8] having a temperature constancy of about one hundredth of a degree in the range 100° to 500°C. In the thermostat core a vacuum chamber consisting of a silica tube (ending about 10 cm outside the thermostat) is mounted. The thermostat and the silica tube can be moved

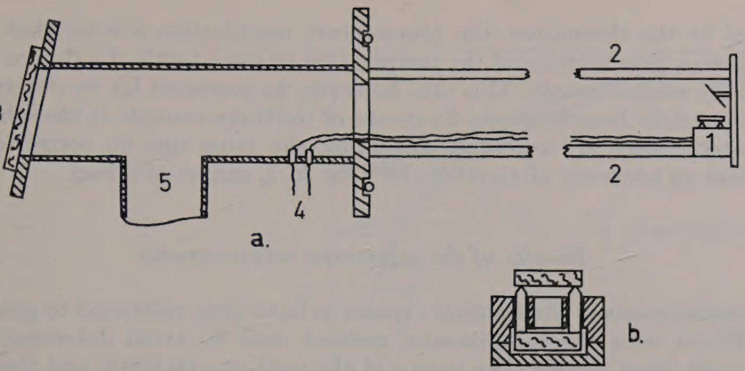

Fig. 6. (*a*) Schematic view of arrangements for the expansion measurements. (*b*) Section of the interferometer.

over the interferometer and the supporting bars. When the interferometer is situated in the middle of the thermostat core, the outer end of the silica tube fits into the rest of the apparatus by means of a rubber gasket and the whole system can be evacuated.

In the interferometer, three pieces of the material whose thermal expansion is to be studied, rest between two optically flat polished silica disks of a diameter of 20 mm. The disks are in the form of a wedge in order to eliminate disturbing interferences. The specimen consists of small rods 3 mm thick and of a height of about 10 mm. The upper end of the rods was cut in the form of a pyramid for providing good contact with the silica plate. The heights of the three specimens were adjusted so as to produce a tilt between the inner surfaces of the disks, which will give rise to 4 to 8 interferences when using the Hg 5461 Å light for illumination. In order to increase the heat capacity of the interferometer it is partly enclosed in a holder of aluminium as shown in Fig. 6*b*. The rods are inserted through carefully polished channels into which they fit loosely in order to avoid disturbing effects from friction between the rods and the aluminium surfaces. In the holder the junction of the thermocouple is located. Immediately above the interferometer a small 45° mirror is mounted for the illumination of the interferometer and for the observation of the interferences through the window of the apparatus.

When starting a series of measurements, the specimen is mounted in the interferometer and rapidly heated to the measuring temperature by placing a small furnace over the interferometer. This heating is done in an inert atmosphere. When the interferometer has reached the measuring temperature the furnace is removed, the thermostat with the silica tube is moved over the interferometer to its inner position, and the apparatus is evacuated. Within one minute after the furnace has been removed the pressure is below 1 μ. After about 5 minutes a final pressure of better than 10^{-2} μ is reached. The linear change of the length of the specimen during the measurements is obtained by recording the number of fringes passing a mark scratched on the surface of the lower silica disk. This is accomplished by photographing the fringe system at frequent intervals. On completion of a run the exposed and developed film is studied under microscope in order to count the number of fringes passing the mark during the run. The fringes can be estimated to one tenth of a fringe or better, corresponding to a relative change of length $\Delta l/l_0$ of the specimens of about 2×10^{-6} where l_0 is the length of the specimen at room temperature. When the interferometer is inserted in the thermostat the temperature distribution is somewhat disturbed, giving rise to a slow increase of the temperature of some tenth of a degree during the course of the measurements. This can, however, be corrected for by determining the temperature of the interferometer by means of the thermocouple at the same moment as the interferences are recorded. Including the error due to corrections of the temperature an accuracy of about 3×10^{-6} for $\Delta l/l_0$ can be obtained.

Results of the expansion measurements

The measurements of the thermal expansion have been restricted to gold as it was found difficult with the experimental method used to avoid deterioration of the specimens for other metals. The purity of the gold was 99.998 % and the grain size about 1 mm, which is the same as for the gold quality denoted I in the calorimetric measurements. The range of measurements was 380° to 480°C. Below 380°C the effect studied was too small to be determined with sufficient accuracy. Above 480°C the temperature constancy of the thermostat was less good.

The dilatation specimen was given a heat treatment which was comparable to that of the calorimetric specimens. It was thus annealed for some days in the thermostat at a given temperature, cooled down in air to room temperature and aged there for two days. Then the specimen was rapidly heated back again to the temperature of the thermostat and the isothermal approach of the thermal expansion to the equilibrium value was studied. It was then found that during about 15 hours a small increase of length of the specimen occured. In Fig. 7 $\Delta l/l_0$ has been plotted versus time for a measurement at 476°C. Here Δl is equal to $(l - l_{eq})$, where l_{eq} is the equilibrium length of the specimen obtained after about 15 hours. No reliable results were obtained until the specimen had reached thermal equilibrium with the thermostat,

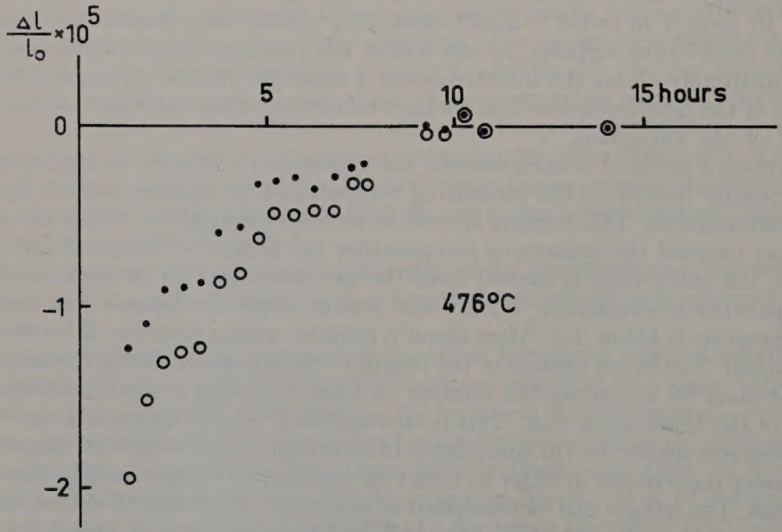

Fig. 7. Relative increase of length versus time for a measurement at 476°C on Au. ○, observed value: ●, corrected value.

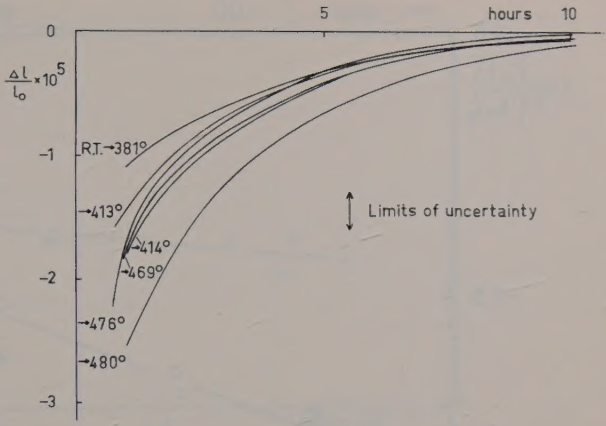

Fig. 8. Relative increase of length versus time for Au at different temperatures.

which took about one hour. The observed values of $\Delta l/l_0$ shown as open circles in Fig. 7, have to be corrected for the drift of temperature of the thermostat and for the change of the refractive index of air with temperature. Under the present experimental conditions the correction of the refractive index is, however, almost negligible. The corrected values are drawn as filled circles. The correction does not exceed 30 %. Within the limits of accuracy it is possible to fit an exponential relation to these values of the form $\Delta l/l_0 = (\Delta l/l_0)_0 \exp(-t/\tau_{\text{exp}})$. The time constant τ_{exp} is about two hours in the present case. The scattering of the observed values is in fact relatively great as a consequence of the small size of the effect studied. It must therefore be emphasised that the use of the exponential representation by no means excludes other possibilities for representation of the observed values. The choice of it may, however, be justified from the results of the comparable calorimetric measurements described in section 2. In Fig. 8 the results are shown in the form of exponential curves for a number of expansion measurements at various temperatures. The extrapolated values of $(\Delta l/l_0)_0$ and the time constants of the curves are collected in Table 2.

Although the scattering of the dilatation measurements is relatively large due to the experimental difficulties, it is clear that the expansion contained in the exponential tail of the isothermal time-curves increases with temperature. The $\log(\Delta l/l_0)_0$ versus

Table 2. Results of the expansion measurements.

T_a °C	T_m °C	$(\Delta l/l_0)_0 \times 10^5$	τ_{exp} h
27	381	1.9	3.5
28	383	1.5	3.2
27	413	2.0	2.8
27	414	2.2	2.9
26	469	2.4	2.7
26	476	2.9	2.2
24	480	3.8	2.8

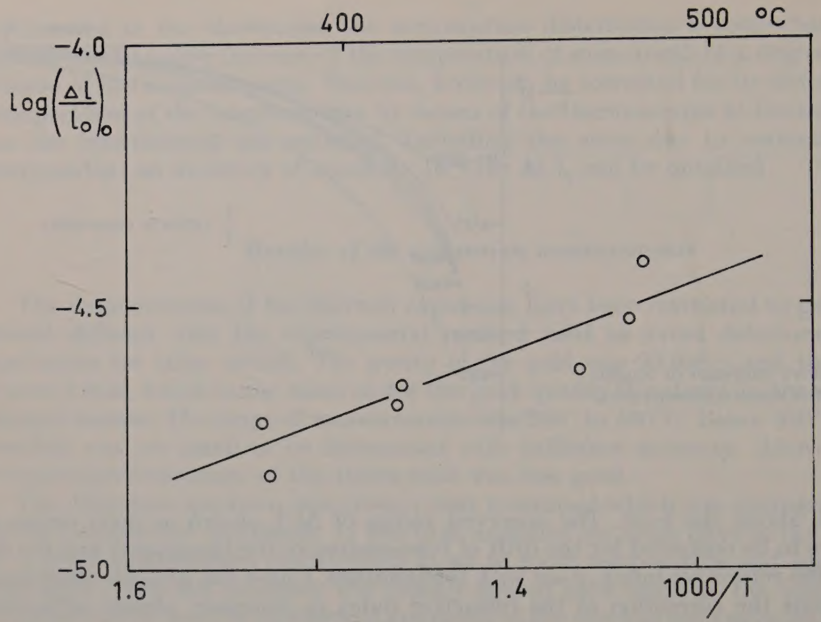

Fig. 9. Logarithms of the relative increase of length versus the reciprocals of the absolute temperatures.

1/*T* diagram in Fig. 9 shows that it is appropriate to express the experimental results in a way similar to eq. (2).

$$\frac{\Delta l}{l_0} = \left(\frac{\Delta l}{l_0}\right)_c \cdot \exp\left[-\frac{H_{\text{exp}}}{R}\left(\frac{1}{T_m} - \frac{1}{650}\right)\right]. \tag{3}$$

The value of the linear expansion at 650°K $(\Delta l/l_0)_c$ is 3.2×10^{-5} and H_{exp} is 6100 cal/mole to within about 30 %.

The temperature dependence of the time constants of the curves in Fig. 8 is shown in Fig. 10. The slope of the line gives an energy of activation of the order 2000 cal/mole but the value is uncertain owing to the inaccuracy in the determination of τ_{exp}. τ_{exp} is, however, of about the same size as τ_{cal}.

4. Discussion of the results

From the above results it seems probable that the isothermally measured increases of energy and volume are closely related to each other. A physical model which will explain the results obtained must then be able to account for the low energy of formation, 2500–6100 cal/mole (0.11–0.26 eV), the weak temperature dependence of the time constants of the rate curves, corresponding to an activation energy of about 1000 cal/mole (0.04 eV) and the fact that the equilibrium value of the energy absorbtion increases with increasing purity (and grain size) of the specimens. Another characteristic feature is the rapid attainment of the equilibrium when the specimens

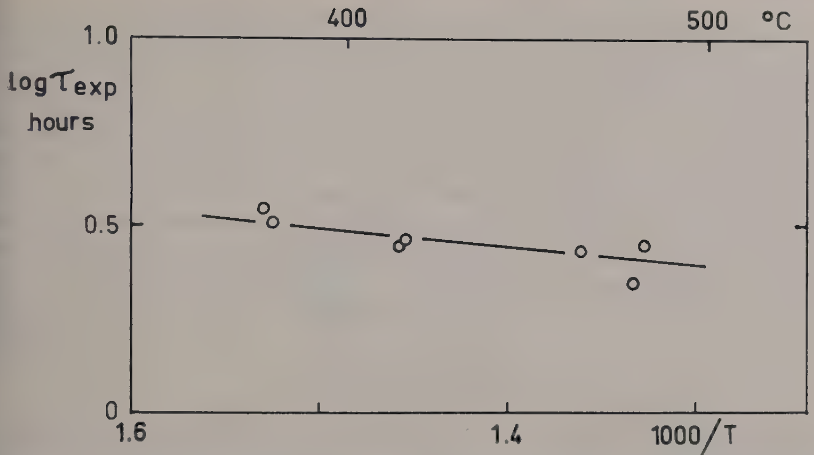

Fig. 10. Logarithms of time constants τ_{exp} versus reciprocals of absolute temperatures for Au.

are quenched in contrast to the rather slow approach when the specimens are heated from a lower ageing temperature to the temperature of observation.

The possibility of explaining the observed phenomena as due to the presence of imperfections in the lattice such as impurities, vacancies and dislocations has been discussed in [1] for the calorimetric measurements on silver. The conclusions arrived at there are still applicable. The high purity of the metals used and the low concentration of thermal vacancies in the actual temperature range makes it improbable that more than a minute part of the observed effects could be due to dissolution of a precipitate or formation of thermal vacancies. Further it is impossible to account for the results by the existence of Cottrell-atmospheres, jogs and/or stacking faults of any known type. It must therefore be assumed that there exists some energy-rich thermal disturbance in the lattice of a type other than has been considered here.

The above results give increasing probability for the assumption that the thermal effects measured in the present case are caused at least partly by the unknown defect which appeared in the phenomenological analysis of the energy content and the thermal expansion by Borelius [3, 4]. The results of these calculations can be expressed in a way similar to eqs. (2) and (3). In Tables 3 and 4 we give the heats of formation for the defects and the increase of energy and volume at 650°K for Au, Ag and Al as obtained from the analysis of Borelius and the corresponding values for the present measurements. For Mo no calculation has been reported but the same scheme would probably be applicable also in this case.

The energies of formation obtained from the calorimetric and the expansion measurements differ somewhat from the phenomenologically derived values for the defects (Table 3) although the difference is almost within the limits of error of the measurements. When comparing the absolute magnitude of the effects (Table 4), however, it is found that the results of the present measurements are only 22 % or less of the calculated values. For gold, where calorimetric and expansion measurements have been made on identical specimens, the ratio between experimental and empirical values, given as Q_c/E_c and $(3\,\Delta l/l_0)_c/(\Delta V/V_0)_c$ respectively, is 0.12 in the first case and 0.08 in the second case. It can, however, be observed that the difference

Table 3. Energy of formation of defects in cal/mole.

(*a*) From analysis of thermal energy content (Borelius).
(*b*) From calorimetric measurements (H_{cal}).
(*c*) From expansion measurements (H_{exp}).

	(*a*)	(*b*)	(*c*)
Au	4530	3300 (I) 3100 (II) ±600	6100 ± 2000
Ag	5160	4200 (A) 4900 (B) 4900 (C) ±700	
Al	4720	3500 ±700	
Mo		2500 ±500	

decreases with increasing purity and grain size of the specimens as shown in the calorimetric measurements on gold and silver. It may therefore be expected that crystals more ideal than those used here would give values nearer to the calculated energies. The reason for the discrepancy may be that the thermal structural disturbances are catalysed by other imperfections in the lattice. This means that for the more imperfect specimens a larger part of the effect may take place too rapidly to be measured with the experimental method used.

From the above discussion it seems probable that the studied thermal effects are caused by the unidentified defects obtained in the analysis of Borelius. This leaves, however, the question of the nature of the observed phenomenon unanswered. The low energy of formation and activation contradicts the possibility that these defects are formed by a reaction which involves diffusion. The extrapolation of the lines in the log $\tau - 1/T$ diagram in Figs. 5 and 10 to $T = \infty$ gives τ-values which are still about 10^{16} times the period of the atomic vibrations. This points to a process of very small thermodynamical probability. It may then be expected that if a reaction is

Table 4.

E_c: Energy of defects in cal/mole at 650°K from analysis of Borelius.
Q_c: Observed absorbed energy in cal/mole at 650°K.
$(\Delta V/V_0)_c$: Increase of volume at 650°K from analysis of Borelius.
$(3\Delta l/l_0)_c$: Observed increase of volume at 650°K.

	E_c	Q_c	Q_c/E_c	$(\Delta V/V_0)_c$	$(3\Delta l/l_0)_c$	$\frac{(3\Delta l/l_0)_c}{(\Delta V/V_0)_c}$
Au	76	8.8 (I) 3.6 (II)	0.12 0.05	1.2×10^{-3}	0.096×10^{-3}	0.08
Ag	79	3.7 (A) 11.4 (B) 17.7 (C)	0.05 0.14 0.22			
Al	105	5.8	0.06			
Mo		5.0				

nevertheless observed to take place with measurable speed at ordinary temperatures, it must be associated with a very small heat of activation (which in fact is obtained). Realization of such a small activation heat seems impossible if we have only processes in which single atoms are activated, but may be possible if groups of atoms are redistributed simultaneously, since the energy per atom for the displacement will decrease with increasing number of atoms in the group. The present measurements do not, however, make it possible to establish a probable model for the thermal defects. It was hoped that the results from Mo, which is body-centered in contrast to the other face-centered metals investigated, would help to throw light upon this question. The energy of formation of the effect obtained for Mo is in fact somewhat lower than for the other metals but the difference is too small to be considered as significant.

SUMMARY

The development of thermal lattice defects has been studied by means of isothermal calorimetry for gold, aluminium and molybdenum of high purity. The specimens, enclosed in argon-filled Pyrex containers, were heated from room temperature to various temperatures in the range 160° to 480°C. The energy absorbed in order to obtain equilibrium at the new temperature had an exponential tail, with a time-constant of a few hours. Increasing temperature leads to an isothermal absorption of heat which becomes larger with increasing purity of the metal. In addition, the thermal expansion in gold has been studied with an isothermal method. With the same pre-annealings as in the calorimetric case a small exponential expansion was observed which took place with about the same rate as the energy absorption. The temperature dependence of the energy and volume increase corresponded to an energy of formation of 0.11–0.26 eV. The time-constants of the experimental curves changed, however, very little with temperature. As impurities, vacancies and/or dislocations cannot explain more than a smaller part of the observed effects, it has been assumed that they may be caused by an unidentified type of thermal defect discussed by Borelius.

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